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13. ABSTRACT (Maximum 200 words)

This research program was aimed at a detailed understanding of the effect of hydrogen as a temporary alloying element (thermohydrogen processing, THP) on processing parameters, microstructural modifications and final mechanical properties of cast-and-wrought (ingot metallurgy), powder metallurgy and cast titanium alloys. Fundamental results have been obtained which can now be used to develop optimum THP steps to refine the microstructure and improve the mechanical properties of titanium alloys. As a part of this project, the phase diagram of the Ti-6Al-4V - hydrogen system, and a determination of the stable and metastable phases, which occur in this system, have been defined. Kinetics of the β phase decomposition has been studied and the non-equilibrium TTT (temperature-time-transformation) diagrams of the beta to alpha+beta+hydride transformation have been determined for the alloys with 10, 20, and 30 at.% hydrogen. Kinetics of decomposition of martensite structures have also been determined for these three hydrogen concentrations. The results have been presented at international meetings, twenty-two technical papers were published and twelve papers have been submitted for publication. Additionally a patent was also awarded. The results of the work have been transitioned to an Army SBIR program designed to produce low cost titanium components for armored vehicles.

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Enhance Performance Near Net Shape Titanium Alloys by Thermohydrogen Processing

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ABSTRACT
This research program was aimed at a detailed understanding of the effect of hydrogen as a temporary alloying element (thermohydrogen processing, THP) on processing parameters, microstructural modifications and final mechanical properties of cast-and-wrought (ingot metallurgy), powder metallurgy and cast titanium alloys. Fundamental results have been obtained which can now be used to develop optimum THP steps to refine the microstructure and improve the mechanical properties of titanium alloys. As a part of this project, the phase diagram of the Ti-6Al-4V - hydrogen system, and a determination of the stable and metastable phases, which occur in this system, have been defined. Kinetics of the β phase decomposition has been studied and the non-equilibrium TTT (temperature-time-transformation) diagrams of the beta to alpha+beta+hydride transformation have been determined for the alloys with 10, 20, and 30 at.% hydrogen. Kinetics of decomposition of martensite structures have also been determined for these three hydrogen concentrations. The results have been presented at international meetings, twenty-two technical papers were published and twelve papers have been submitted for publication. Additionally a patent was also awarded. The results of the work have been transitioned to an Army SBIR program designed to produce low cost titanium components for armored vehicles.

BACKGROUND
Titanium and conventional titanium alloys have a high affinity for hydrogen and are capable of absorbing up to 60 at.% hydrogen at 600°C and even higher contents can be alloyed with titanium at lower temperatures [A]. The fact that hydrogen can be added by exposing the titanium samples to a hydrogen environment at elevated temperatures and can then be easily removed by a simple vacuum annealing makes it a unique temporary alloying element [A-L]. Hydrogen as an alloying element changes the phase compositions and kinetics of the phase transformations in titanium alloys, allowing novel thermal and thermomechanical treatments. As a result, novel microstructures and enhanced mechanical properties can be obtained after the thermohydrogen processing (THP) [A-L]. At sufficiently high hydrogen concentrations, room temperature embrittlement provides an economic method for production of titanium powder (the hydride-dehydride, HDH, process), with the hydrogen then removed by a simple vacuum anneal. Temporary alloying of hydrogen with Ti-6Al-4V, the most popular α+β alloy which accounts for more than half the total titanium sales, has been of interest in a number of investigations [J-R], because improved workability and refined microstructures can be achieved, which are not possible otherwise [A,J-L,S, T].
Improvement in superplastic forming of Ti-6Al-4V has been achieved by temporary alloying with hydrogen [M,Q,U]. A reduction of 30% in flow stress or lowering in forging temperature by 80°C using 13-16 at.% hydrogen has been reported by Kerr et al. [J]. Zwicker et al. have also reported improvement in forgeability of titanium alloys when temporarily alloyed with hydrogen [V]. Ultrafine equiaxed grains can also be produced in Ti-6Al-4V by temporary alloying with hydrogen leading to improvement in mechanical properties [K,O]. Although the benefits of temporary alloying of Ti-6Al-4V with hydrogen are numerous, the phase transformations in the system have not been studied extensively. Ti-6Al-4V - Hydrogen phase diagrams have been suggested by Kerr et al. [J] and Ilyn et al. [L], which are essentially different. Based on their OM and XRD analyses, Kerr et al. [J] have proposed a hydride phase formation by an eutectoid transformation of the beta phase near 800°C, a considerable departure from the pure titanium-hydrogen system in which the eutectoid transformation occurs 500°C lower at 300°C [W]. On the other hand, Ilyn et al. [L], have suggested the phase diagram with a hydride phase present only below 300°C and a wide α+β phase range present above 300°C. It was clear that detailed additional study was required to determine the correct equilibrium phase diagram.
In order to fully define the effect of heat treatment on the hydrogenated alloy, non-equilibrium TTT-diagrams are required at different hydrogen concentrations. However, only one TTT diagram for beta-phase decomposition (β-TTT diagram) is available, that for the alloy with 40 at.% hydrogen [J]. However the authors themselves [10] have questioned the validity of the results.
The present project was aimed at a detailed understanding of the effect of hydrogen as an alloying element on equilibrium and non-equilibrium phase transformations and microstructural evolution in various Ti-6Al-4V product forms. The Ti-6Al-4V - Hydrogen phase diagram was determined using optical microscopy, transmission electron microscopy and X-ray diffraction techniques and compared with previously suggested diagrams. The non-equilibrium β-TTT diagrams were also determined for the alloy with 10, 20 and 30 at.% H. Martensite decomposition behavior for 10, 20 and 30at.% H containing alloys have also been studied.

RESULTS
During the period of this research work the following tasks were completed.

1. Microstructures, phases and phase transformations in the Ti-6Al-4V alloy containing 0.0 at.% [base], 10at.%, 20at.% and 30at.% hydrogen were investigated using optical microscopy (OM), transmission electron microscopy (TEM), X-ray diffraction (XRD), differential thermal analysis (DTA) and microhardness testing. Special heat treatments (with an increasing annealing time and/or decreasing temperature) were conducted on the hydrogenated specimens to determine the phase boundaries in the Ti-6Al-4V-xH system in the temperature range of 20°C to 1000°C and kinetics of the beta phase decomposition evaluated. A currently available Ti-6Al-4V-xH phase diagram was considerably modified and improved. In these temperature-concentration ranges studied three

stable phases were identified: hcp alpha, bcc beta, and fcc delta (hydride). The beta transus temperature of Ti-6Al-4V alloy decreases with an increase in the hydrogen concentration from 1005°C at 0% H to 815°C at 30% H. The decrease is rapid at concentrations less than 10%H and much smaller down at higher hydrogen concentrations. A two-phase alpha+beta field is present at hydrogen concentrations of 0-10 at.% below the beta transus. A three-phase alpha+beta+delta field is present at hydrogen concentrations above 10 at.% and below the beta transus. A two phase beta +delta field is present at hydrogen concentrations of □ 20 at.%, above the beta transus and below 900°C. TTT diagrams for isothermal beta phase decomposition were determined for the Ti-6Al-4V alloy containing 10, 20 and 30 at.% hydrogen. The beta phase decomposed into alpha, beta and hydride phases. It was found that the addition of hydrogen stabilized the beta phase hence increasing the time required to begin and end the decomposition. Alloying with hydrogen increases the nose time and decreases the nose temperature of the TTT start curves. The nose time increases from 12 seconds to 42 minutes and the nose temperature decreases from 725°C to 580°C when the hydrogen concentration increases from 0 to 30 at.%. The minimum cooling rate for 100% martensite transformation decreases from about 1000°C/min to 5°C/min and the maximum cooling rate below which no martensite transformation occurs decreases from about 30°C/min to 1°C/min when the hydrogen concentration increases from 0 to 30 at.% Based on these results, four papers [1,15-17] have been submitted and accepted for publication. The results have been presented at various international conferences [35-38].

2. Kinetics of the martensite phase decomposition in the hydrogen containing alloys have also been determined. Hexagonal α' and orthorhombic α'' martensites are formed in the hydrogenated Ti-6Al-4V alloy after water quenching from the beta phase field; with the volume fraction of the α'' martensite increasing from 0 to 80% when the hydrogen concentration increases from 0 to 30 at.%. A small amount of a δ hydride phase is also detected in the alloys with 20 at.%H and 30 at.%H. Hardness of the martensite structure decreases when the hydrogen concentration is increased as a result of an increasing volume fraction of the softer α'' martensite. During aging at a temperature below the beta transus, the martensite structure transforms into stable alpha and beta phases. The kinetics of the martensite decomposition depends on the aging temperature and the hydrogen concentration. The temperature at which the rate of the decomposition is the fastest (the nose temperature) decreases, while a transition time required to start the transformation at this temperature (the nose time) does not change significantly when the hydrogen concentration is increased. Complete decomposition of the martensite structure results in a microstructure consisting of a mixture of elongated and equiaxed microstructure in Ti-6Al-4V alloy with 20 at.% H and in a fine equiaxed microstructure in Ti-6Al-4V alloy with 30at.% H. Hydrogen alloying has lowered the M_s temperature of the Ti-6Al-4V alloy from ~900°C for 0% H to below 530°C for 30at.% H. Papers based on these results have been submitted for publications [2,18]. The results will also be presented in an international conference [39].

3. Hydrogen was employed as a temporary alloying element in the mechanical alloying (MA) process to produce novel titanium alloys and microstructures with enhanced properties. The presence of hydrogen in powders during mechanical alloying enhanced fracturing, refinement, and mixing of the powder particles, accelerated transition of material to an amorphous state, speeded up phase reactions during post-MA heat treatment, and reduced contamination from oxygen. Ti-Al, Ti-Al-Si, and Ti-Mg-Si alloys, intermetallics and composites with submicrocrystalline structures were produced by mechanical alloying of corresponding pre-alloyed and/or elemental powders and the titanium hydride followed by hot isostatic pressing (HIP'ing) and hydrogen removal, and microstructures of these materials were evaluated using TEM, SEM, XRD, and DTA. These alloys are attractive for high temperature applications, however their use is limited by poor ambient temperature ductility. Microstructural refinement via far-from-equilibrium processing methods, ternary alloying additions and proper thermal treatment can improve the mechanical property combination. The results have published [3-6,19-23] and presented in international meeting [40-42].

4. Synthesis of Ti_3SiC_2 was also attempted via mechanical alloying using titanium hydride as the starting material. Two different powder mixtures were used to synthesize Ti_3SiC_2 (a ductile ceramic). One powder mixture comprised of titanium hydride and SiC. Another powder mixture used contained titanium hydride along with silicon and graphite powder. In either case the powder mixture was mechanically alloyed (MA) for five hours. The mechanically alloyed powder mixture was hot isostatically pressed (HIP'd) at 1200°C. MA powders and the HIP'd samples were further studied using optical microscopy, SEM, TEM, DTA and XRD. X-ray diffraction patterns of the HIP'd sample showed peaks corresponding to $Ti3SiC_2$. Also, seen in the X-ray diffraction pattern were peaks representing TiC. So as per the present findings MA offers an alternate method for producing $Ti3SiC_2$.
In an attempt to produce a low-density titanium alloy starting with titanium hydride, powder mixture containing titanium hydride, magnesium and boron was mechanically alloyed. MA powder mixture was later HIP'd at 800°C. MA powder as well as the HIP'd alloy was studied using optical microscopy, SEM, TEM, XRD and DTA. HIP'd sample showed a uniform distribution of TiB_2 phase in titanium matrix. The HIP'd sample with dispersed TiB_2 is expected to have a low density and improved mechanical properties.

5. The steady-state plastic flow of a series of beta Ti-H alloys containing up to 31 at.% hydrogen was analyzed using the theory of thermally activated dislocation glide. It was found that the pre-exponential factor decreased and the Gibbs free energy of the plastic flow increased when the hydrogen concentration was increased owing to the strengthening effect of hydrogen on the shear modulus of beta titanium. It is concluded that the steady-state flow of beta Ti-H alloys is controlled by a single thermally activated mechanism and that the observed hydrogen-induced hardening is largely caused by the stiffening effect of hydrogen on the shear modulus. Results were published in Philosophical Magazine [7].

6. Neutron diffraction experiments were conducted at the DOE ORNL facility in May-June 2000 to study the effect of hydrogen on the lattice parameter and thermal expansion of the beta titanium. The solid solution of hydrogen in beta titanium expanded the lattice but had little effect on the thermal expansion coefficient. The dependence of the lattice parameter on atomic percent of hydrogen was found to be linear over the hydrogen concentration range of 0 to 30 at.%. A reversible crystalline-amorphous state transformation was detected in highly hydrogenated alloys near 800°C. The results were published in a paper [8].

7. Production of low cost titanium parts from titanium turnings using hydrogen as a temporary alloying element was investigated. This work has additionally been supported by the US army research laboratory, contract # DAAD-17-99-C0022. Commercially pure (CP)

titanium and Ti-6Al-4V powders with allowable oxygen and nitrogen content were successfully produced from their respective turnings. The results were published in several papers [24-32] and presented at international conferences [42-48]. This work was conducted together with ADMA Products, Inc., Twinsburg, OH, and led to additional funding from the US ARL with SBIR Phase II program funding. Currently, production of fully dense large compacts from the powders is underway for initial mechanical and ballistic testing.

8. Several review papers containing information on thermohydrogen processing and describing the results obtained within the research program were prepared and published during the period reported [10-13, 33].

Three senior personnel (Drs. F.H. (Sam) Froes, O.N. Senkov and S.N. Patankar), three graduate students (J.I. Qazi, J. Rahim and M. Cavusoglu) and two undergraduate students (M. Schnider and C. Marshall) have worked on this project. A major part of this research work will become part of Mr. J.I. Qazi's Ph.D. dissertation and Mr. J. Rahim's MS thesis. Working within the project the graduate students received the following awards:

Javaid I. Qazi
- 2nd Position University of Idaho GSA Research Exhibition, 2001.
- 2nd position 2001 Annual Optical Microscopy competition, Inland Empire Chapter of American Society of Metals (ASM).
- Outstanding service award, Dept. Mat. Science, University of Idaho, 2000.
- 1st Prize TMS Student Poster Competition, TMS 2000 Annual Meeting, Nashville, TN.
- Graduate Student Association (GSA) Travel Award to attend Aeromat'2000 Bellevue, WA..
- GSA Travel Award to attend TMS annual meeting 2000, Nashville, TN.
- Alumni Award of Excellence, University of Idaho, 1999.
- TMS International Structural Material Division (SMD) Travel Award for the TMS Fall Meeting 1999.
- One of the world's four recipients for the Center for Powder Metallurgy Technology (CPMT)/ Axel Madsen Award travel award, 1999.
- Recipient of the International Student Scholarship Endowment Award, University of Idaho, Fall 1999.
- 2nd position in the student category of the 1999 International Powder Metallurgy Metallography Competition, $PM^2TEC'99$.
- GSA travel award to attend the TMS annual meeting 1999, San Diego, CA.
- 1st position 99 Annual Optical Microscopy competition, Inland Empire Chapter of ASM.

Mutlu Cavusoglu
Masters Degree, May 2000
United Engineering Foundation, Inc., Conference Fellowship; 1999.
Graduate Student Association, University of Idaho Travel Award, February 1999
Graduate Student Association, University of Idaho Travel Award, September 1999.
Graduate Student Association Research Exhibition/Poster Competition Award, April 1999.
Graduate Student Association Research Exhibition/Poster Competition Award, April 2000.

Jawad Rahim
1st Position in ASM Inland Empire photomicrograph Competition, April 10, 2001

PUBLICATIONS

Peer Reviewed Journals

1. J.I. Qazi, O.N. Senkov, J. Rahim, A. Genc and F.H. (Sam) Froes, Phase Transformations In Ti-6Al-4V-xH Alloys, *Metal. Mat. Trans. A*, 2001, Vol. 32A, (10) pp. 2453-2463.
2. J.I. Qazi, J. Rahim, O.N. Senkov and F.H. (Sam) Froes, Kinetics of Martensite Decomposition in Ti-6Al-4V-xH System, to be submitted.
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4. M. Cavusoglu, O.N. Senkov, F.H. Froes, M.L. Ovecoglu, T. Zimmerly and O. Inal, Compaction of Nanocrystalline Gamma-Titanium Aluminides, *Key Eng. Mat.*, 2000 Vol. 188, pp. 15 –26.
5. M. Cavusoglu, O.N. Senkov and F.H. Froes, Synthesis and Characterization of a Low Density Ti-Mg-Si Alloy, *Key Eng. Mat.*, Vol. 188, 1 –14.
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8. O.N. Senkov, B.C. Chakoumakos, J.J. Jonas, and F.H. Froes, Effect of Temperature and Hydrogen Concentration on the Lattice Parameter of Beta Titanium, *Mater. Research Bulletin,* 2001, Vol. 36/7-8, pp. 1433-1442.
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10. F.H. Froes, O.N. Senkov, and E.G. Baburaj, Synthesis of Nanocrystalline Materials – an Overview, *Mat. Sci. Eng. A*, 2001, Vol. 301 (1), pp. 44-53.
11. D. Eliezer, N. Eliaz, O.N. Senkov, and F.H. Froes, Positive Effects of Hydrogen in Metals, *Mat. Sci. Eng. A*, 2000, Vol. 280A (1), pp. 220-224.
12. F.H. Froes, O.N. Senkov and J.I. Qazi, Hydrogen as a Temporary Alloying Element in Titanium Alloys, to be published *Inter. Mat. Rev.* 2002.
13. O.N. Senkov and F.H. Froes, Thermohydrogen Processing of Titanium Alloys, *Inter. J. Hydrogen Energy*, 1999, Vol. 24 (6), 565-576.
14. J.I. Qazi, J. Rahim, O.N. Senkov and F.H. (Sam) Froes, Microstructure Evolution in Ti-6Al-4V-xH System, to be submitted.

Non Peer Reviewed Journals and Conference Proceedings

15. J.I. Qazi, O.N. Senkov, J. Rahim, S.N. Patankar and F.H. (Sam) Froes, Phase Transformations In Ti-6Al-4V-xH Alloys, *JOM*, accepted for publication.
16. J.I. Qazi, O.N. Senkov, J. Rahim, S.N. Patankar and F.H. (Sam) Froes, Phase Transformations In Ti-6Al-4V-Hydrogen System, to be published in *Proceedings of the materiais2001, April 9-11,* Combira Portugal.
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24. V.S. Moxson an F.H. (Sam) Froes, Fabricating Sports Equipment Components via Powder Metallurgy, *JOM*, 2001, Vol. 53, no. 4, pp. 39-41.
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26. V.S. Moxson, O.N. Senkov, and F.H. Froes, Innovations in Titanium Powder Processing, *JOM*, 2000, Vol. 52 (5), pp. 24-26.
27. J.I. Qazi, V.S. Moxson, O.N. Senkov and F.H. Froes, Recycling of Titanium and Ti-6Al-4V Turnings Using Thermohydrogen Processing, in: R.D. Peterson (Ed.), *Light Metals 2000*, TMS, Warrendale, 2000, pp. 885-889.
28. V.S. Moxson, J.I. Qazi, S.N. Patankar, O.N. Senkov and F.H. (Sam) Froes, Low Cost CP-Titanium and Ti-6Al-4V Alloys, to be published in *Proceedings of the materiais2001, April 9-11,* Combira Portugal.

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PRESENTATIONS AT THE INTERNATIONAL CONFERENCES

35. J.I. Qazi, O.N. Senkov, J. Rahim, S.N. Patankar and F.H. (Sam) Froes, Phase Transformations In Ti-6Al-4V-Hydrogen System, in: *materiais2001, April 9-11,* Combira Portugal.
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40. M. Cavusoglu, J.I. Qazi, O.N. Senkov and F.H. (Sam) Froes, Synthesis and Microstructure of Low Density Ti-Si-Al and Ti-Mg-Si Alloys, *Annual Advanced Aerospace Materials and Processes Conference, June, 26-29, 2000 Bellevue, WA.*
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